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KEL-F[®] ELASTOMER
"A NEW MATERIAL FOR THE ELECTRICAL INDUSTRY"

by

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This paper is designed to acquaint the people in the wire and cable industry with a new elastomer which we believe can be of assistance in the design of improved equipment for generating, conveying, transforming, and utilizing electrical energy.

Toward this end, we will here summarize the compounding and significant properties of KEL-F Elastomer as related to its potential application in the electrical industry.

This new polymer, called KEL-F Elastomer, was developed by The M. W. Kellogg Company in cooperation with the Office of the Quartermaster General, U. S. Army. This elastomer which has been undergoing further development by the Kellogg Company is expected to be in semi-commercial production in March of 1955.

As can be seen in our first slide, KEL-F Elastomer is a saturated fluorocarbon polymer containing more than 50 per cent fluorine by weight. It will be available as either a white spongy crumb or a water dispersion. The specific gravity of the gum is approximately

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1.85. It is readily soluble in ketones, esters and cyclic ethers, but insoluble in alcohols and aliphatic, aromatic and chlorinated hydrocarbons.

KEL-F Elastomer in the uncured state has a tensile strength of 300-600 p.s.i., an elongation of 600-800 per cent and a durometer hardness of 40-45A. The raw gum has excellent storage life. It is amorphous unstretched, but orients upon stretching to give a typical X-ray fiber diagram.

Although the gum has inherently good physical characteristics, it must be vulcanized to obtain the ultimate in physical properties, heat resistance and chemical resistance.

Because of its completely saturated chain structure, KEL-F Elastomer cannot be vulcanized with sulfur by the usual methods. However, the polymer can be vulcanized by organic peroxides, polyisocyanates, polyamines, and isocyanate amine combinations. These curing agents when accelerated by metallic oxides such as those of zinc, calcium, lead, and magnesium will cross link the gum to form strong extensible gum stocks having tensile strengths up to 2500 p.s.i. and elongations of 400-600 per cent. KEL-F Elastomer may, also, be cross linked by irradiation from a gamma ray source.

Slide II illustrates a typical example of each method of vulcanization.

Undoubtedly, each vulcanization system will ultimately find specific application. However, due to the excellent overall characteristics of the peroxide method of vulcanization, we shall emphasize

this system in today's discussion.

In stocks cured with organic peroxides, basic lead salts, such as dibasic lead phosphite, tribasic lead maleate and tribasic lead sulfate are used as secondary accelerators to improve and maintain the physical properties. This is graphically shown in Slide III.

In addition to gum stocks, it is possible to compound KRL-F Elastomer with various fillers, a few of which reinforce the stock resulting in higher tensile strengths, higher moduli, and increased hardness, and tear strength. Slide IV shows a group of filled stocks which includes 3 silicas, a metal silicate and a furnace black. Stock 122 is of special interest because of the extreme reinforcement in ultimate tensile strength obtained from a filler made from a precipitated silica, Hi-Sil C, which has been coated with a low molecular weight silicone polymer. Conversely, uncoated Hi-Sil C increases modulus and hardness with no effect upon tensile strength. A highly refined silica, Hi-Sil 303, reinforces the stock to give excellent physical properties including very high tear strength. In addition, the thermal stability of this stock is excellent. Many of the metal silicates, such as the zirconium silicate, and Asbestine 3X, the clays such as Iceberg Pigment, and other fillers such as barium sulfate, and anhydrous alumina have been tested in peroxy cured stocks. These fillers function

more as extenders and have little or no effect upon tensile strength. In addition, these stocks tend to degrade during prolonged exposure at 400°F. To date, we have had little success in utilizing C-black in the peroxide cure due to poisoning of the vulcanizing reaction accompanied by excessive decomposition and blowing. Conversely, the blacks have been used successfully to reinforce both the polyisocyanate and polyamine cures.

Much remains to be done to fully define the application of fillers, especially where dielectric properties are involved.

Now that we have examined the compounding and physical properties of KEL-F Elastomer, let us examine some of its other properties.

Due to its high fluorine content, KEL-F Elastomer is quite inert and, consequently, chemical and heat resistant, non flammable, fungi resistant and abrasion resistant.

Like its precursor, the most outstanding characteristic of KEL-F Elastomer is its chemical resistance. It is resistant to strong oxidizers, mineral acids, peroxides, alkalies, some chlorinated solvents, alcohols and aliphatic solvents. It is also resistant to many hydraulic fluids and lubricants such as the sulfur bearing or halogen bearing extreme pressure lubricants, as indicated in Slide V.

In Slide VI, we have a rather dramatic illustration of the inertness of KEL-F Elastomer as compared with conventional rubbers after immersion in white fuming nitric acid. KEL-F Elastomer reaches an equilibrium swell of 25% after one week.

With the operating temperatures of electrical equipment mounting every day, it might be well to look at the thermal stability of KEL-F Elastomer. First, we might examine the effect of hot paraffin oil and an extreme pressure lube oil on KEL-F Elastomer as shown in Slide VII. Additional data show KEL-F Elastomer to be extremely resistant to silicone oil, DC-200, and chlorinated polyphenyls, Arochlor 1254 at 300°F.

Slide VIII shows the retention of tensile properties after 30 days conditioning in circulating air ovens at 300°, 400°, and 480°F. Note that continued exposure at 300°F. increases tensile strength without shortening the stock. At 400°F., 75% of tensile strength is retained, with elongation and hardness unchanged. Continued exposure at 480°F. causes degradation of the network. Slide IX again shows these relations wherein the rate of change of tensile strength is plotted.

Along with the vulcanizates, the thermal stability of the gum at 400°F. is excellent. Prolonged exposure shows no evidence of chain breakdown or loss of halogen.

In addition to its thermal stability in air, the vulcanizates show good resistance to high pressure steam. After 7 days exposure at 247 p.s.i.a. and 400°F., the 89 stock retained 80% of its tensile strength with no loss in elongation. The volume swell under these conditions was 22%.

During the early days of its evaluation, it was evident that KEL-F Elastomer did not meet the specifications for an arctic rubber. I would like to discuss this limitation, and some of the approaches being taken to solve this problem.

Slide X shows the low temperature properties of the vulcanizates. As with the fluorocarbon thermoplastics, the embrittlement point is low. However, as shown here in the 89 stock, the stiffness increases rapidly over the range 0 to -10°C. The hardness increases correspondingly over this range. We are currently doing work on this problem by two approaches, namely:

1. We are endeavoring to increase chain mobility by changing polymer structure.
2. We are compounding the elastomer with high-boiling hydrocarbon esters. Such esters, when added to the polyamine recipe, produced a marked improvement in the stocks low temperature properties, shifting the curve of stiffness versus temperature from 0/-10°C. to -30/-40°C. The resultant stock also showed improved resilience and compression set. As you can see, there still

remains plenty of room for those skilled at compounding to apply their skill to this material.

On Slide XI, we have the resistance of KEL-F Elastomer to some of the more common enemies of all electrical insulation; namely, oxygen, ozone, ultraviolet radiation, and weathering.

In the same vein, Slide XII shows one of the characteristics which I am sure will be of interest to all concerned with insulation. The water absorption of KEL-F Elastomer is compared with the values for rubbers currently in commercial use.

Slide XIII illustrates our major point of interest here today, the electrical values of KEL-F Elastomer. For many of you, these values may appear similar to those obtained from a vinyl compound. However, one significant difference may be seen when we note the relatively slight change in power factor before and after conditioning of 95% relative humidity. Such values are indicative of the low moisture absorption noted for KEL-F Elastomer. KEL-F Elastomer is compatible and has been vulcanized in combination with silicone rubber, neoprene, GR-N, GR-S, Hypalon, and butyl rubbers.

In terms of practical application, KEL-F Elastomer has been compounded, molded, extruded, calendered, and coated

on standard rubber equipment.

Slide XIV shows the forms of raw material available for compounding and fabrication:

1. low-solids latex.
2. the gum in the form of a spongy crumb and sheeted stock.

Slide XV shows some of the types of articles fabricated by compression molding. These include: sheets, "O" rings, and unsupported and reinforced diaphragms. Not shown here are grommets for electrical connectors. In general, one can state that intricate parts can be compression or transfer molded without difficulty.

Slide XVI shows two coils of tubing extruded on a 1 inch NRM bench extruder. This tubing has a tensile strength greater than 1500 p.s.i., and elongation of 400%. The 1/4 inch I.D. tubing having a thickness of 0.035 inches, proved to have a dielectric strength of 16.5 KV or 470 volts per mil.

KEL-F Elastomer has been dissolved in mixtures of ketones and esters to form smooth cements. Diluents such as toluene and n-butanol may be added to lower viscosity. These cements have been applied by spreading and dipping to various fabrics and forms. Slide XVII shows an unsupported glove and a section of coated nylon produced from KEL-F Elastomer cement. Resistance measurements on a section of coated nylon having a film thickness of .003 inches

showed a resistivity of 7×10^4 megohms at 500 volts.

Much remains to be done in applying and testing KEL-F Elastomer as an electrical insulation designed to meet one's particular specification. As a raw material, we do believe it comes equipped with many excellent properties required for such service. These include: high tensile strength, extensibility, tear strength, abrasion resistance, thermal stability, extremely low water absorption, and resistance to oxidants, and a variety of chemicals.

It is hoped that these characteristics will assist the electrical industry in the design of specialized insulation to be used in those applications where a high strength rubber must withstand heat and corrosive chemicals. Such applications immediately foreseen are insulated wire and cable, sheath stock, tapes, potting compounds, and electrical connectors, grommets, and seals.

SLIDES

- I KEL-F Elastomer Gum
- II Vulcanization of KEL-F Elastomer
- III Effect of Lead Salts and Metallic Oxides
- IV Filler Study
- V Chemical Resistance
- VI Picture of White Fuming Nitric Acid Resistance
- VII Hot Oil Resistance of KEL-F Elastomer
- VIII Thermal Stability of KEL-F Elastomer
- IX Thermal Stability of KEL-F Elastomer (Tensile vs. Time)
- X Low Temperature Properties
- XI Aging Characteristics of KEL-F Elastomer
- XII Water Absorption
- XIII Electrical Values
- XIV Picture of Raw Materials
- XV Picture of Compression Molded
- XVI Picture of Extruded Tubing
- XVII Picture of Glove and Coated Fabric

SLIDE I

KEL-F ELASTOMER GUM

SPECIFIC GRAVITY	1.85
FLUORINE CONTENT	> 50%
COLOR	TRANSLUCENT, OFF-WHITE
TENSILE, psi	300-600
ELONGATION, %	600-800
SHORE A HARDNESS	40-45
INTRINSIC VISCOSITY, 0.5% MEK 30°C.	2-3
SOLUBILITY (25% CEMENTS)	KETONES, ESTERS, ETHERS
STORAGE	> 2 YEARS

SLIDE II

VULCANIZATION OF KEL-F ELASTOMER

<u>CURATIVE TYPE</u> <u>STOCK NUMBER</u>	<u>PEROXIDE</u> <u>61</u>	<u>AMINE</u> <u>37</u>	<u>MDI</u> <u>1</u>	<u>MDI-AMINE</u> <u>98</u>
KEL-F ELASTOMER	100	100	100	100
ZINC OXIDE	10	5	5	5
BENZOYL PEROXIDE	3	-	-	-
MDI-100	-	-	5	5
TEPA	-	6	-	1
<u>CURE</u>				
PRESS HR./°F.	1/2/230	1/260	1/260	1/260
OVEN HR./°F.	16/300	1/300	72/212	16/212
STRESS AT 300% psi	530	1020	590	630
TENSILE, psi	1350	1620	800	1250
ELONGATION, %	500	450	320	580
HARDNESS, SHORE A	47	55	61	60

SLIDE III

BASIC LEAD SALTS AND METALLIC OXIDES
EFFECT ON A PEROXIDE CURED KEL-F ELASTOMER

<u>STOCK NUMBER</u>	<u>61</u>	<u>89</u>	<u>108</u>	<u>111</u>
KEL-F ELASTOMER	100	100	100	100
ZINC OXIDE	10	10	-	10
LITHARGE	-	-	10	-
BENZOYL PEROXIDE	3	3	3	3
DIBASIC LEAD PHOSPHITE	-	10	10	-
TRIBASIC LEAD MALEATE	-	-	-	10
<u>CURE</u>				
PRESS HR./°F.	-----	1/2/230	-----	-----
OVEN HR./°F.	-----	16/300	-----	-----
STRESS AT 300%, psi	530	660	435	490
TENSILE, psi	1350	2500	2200	2400
ELONGATION, %	500	500	650	550
HARDNESS, SHORE A	47	51	48	47

SLIDE IV

FILLED KEL-F ELASTOMER

<u>STOCK NUMBER</u>	<u>119</u>	<u>122</u>	<u>183</u>	<u>160</u>	<u>166</u>
KEL-F ELASTOMER	100	100	100	100	100
ZINC OXIDE	10	10	10	10	10
DIBASIC LEAD PHOSPHITE	10	10	10	10	10
BENZOYL PEROXIDE	3	3	3	3	3
HI SIL-C	20	-	-	-	-
SILICONE COATED					
HI SIL-C (LM-3)	-	20	-	-	-
HI SIL X-303	-	-	20	-	-
ZIRCONIUM SILICATE	-	-	-	20	-
PHIL BLACK-A	-	-	-	-	20

CURE

PRESS: 1/2 HR./230°F.

OVEN: 16 HR./300°F.

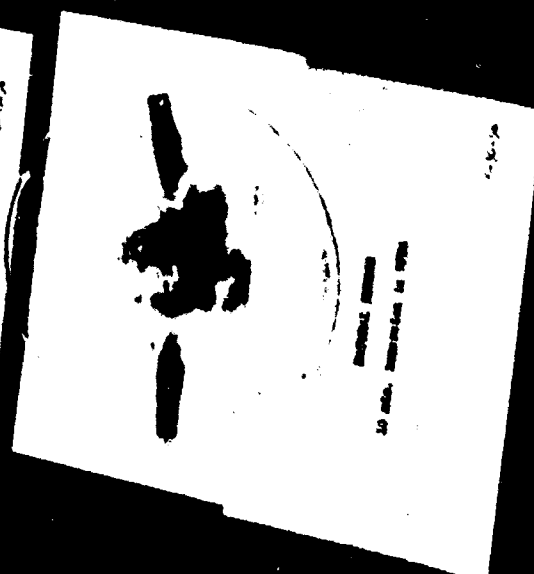
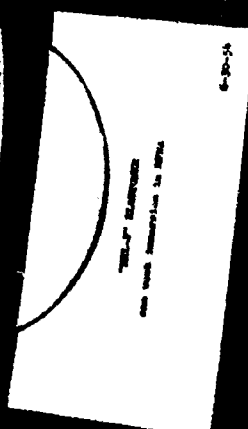
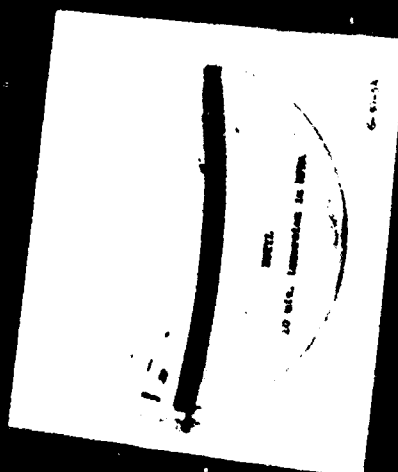
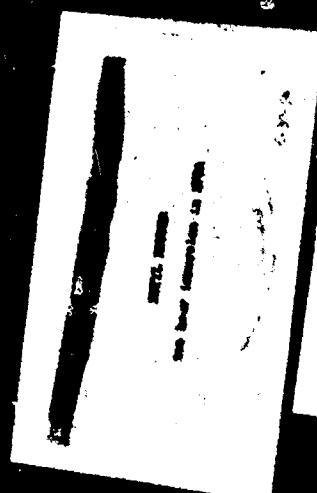
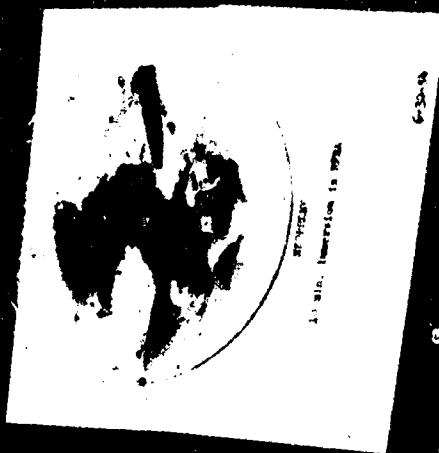
STRESS AT 300%, psi	1230	1620	1200	800	290
TENSILE, psi	1880	3600	2700	2510	420
ELONGATION, %	525	450	660	700	450
SHORE A HARDNESS	70	73	75	60	69

SLIDE V

CHEMICAL RESISTANCE
PEROXIDE CURED KEL-F ELASTOMER

DAYS:	% VOL. SWELL. 70°F.		
	<u>1</u>	<u>2</u>	<u>27</u>
<u>ACIDS</u>			
WHITE FUMING NITRIC	12	22	24
RED FUMING NITRIC	9	15	24
FUMING SULFURIC	0	0	1
12M HYDROCHLORIC	3	3	16
<u>SOLVENTS</u>			
PETROLEUM ETHER	5	5	13
ANILINE	0	1	5
ETHYL ALCOHOL	2	2	6
TOLUENE	40	60	60
CARBON TETRACHLORIDE	15	30	33
<u>FUELS-LUBRICANTS</u>			
ISOOCTANE-TOLUENE 70/30	-	-	16
E. P. LUBE (Elco-28)	0	0	1
DI-ESTER (TURBO-OIL-15)	16	24	600

SLIDE VI



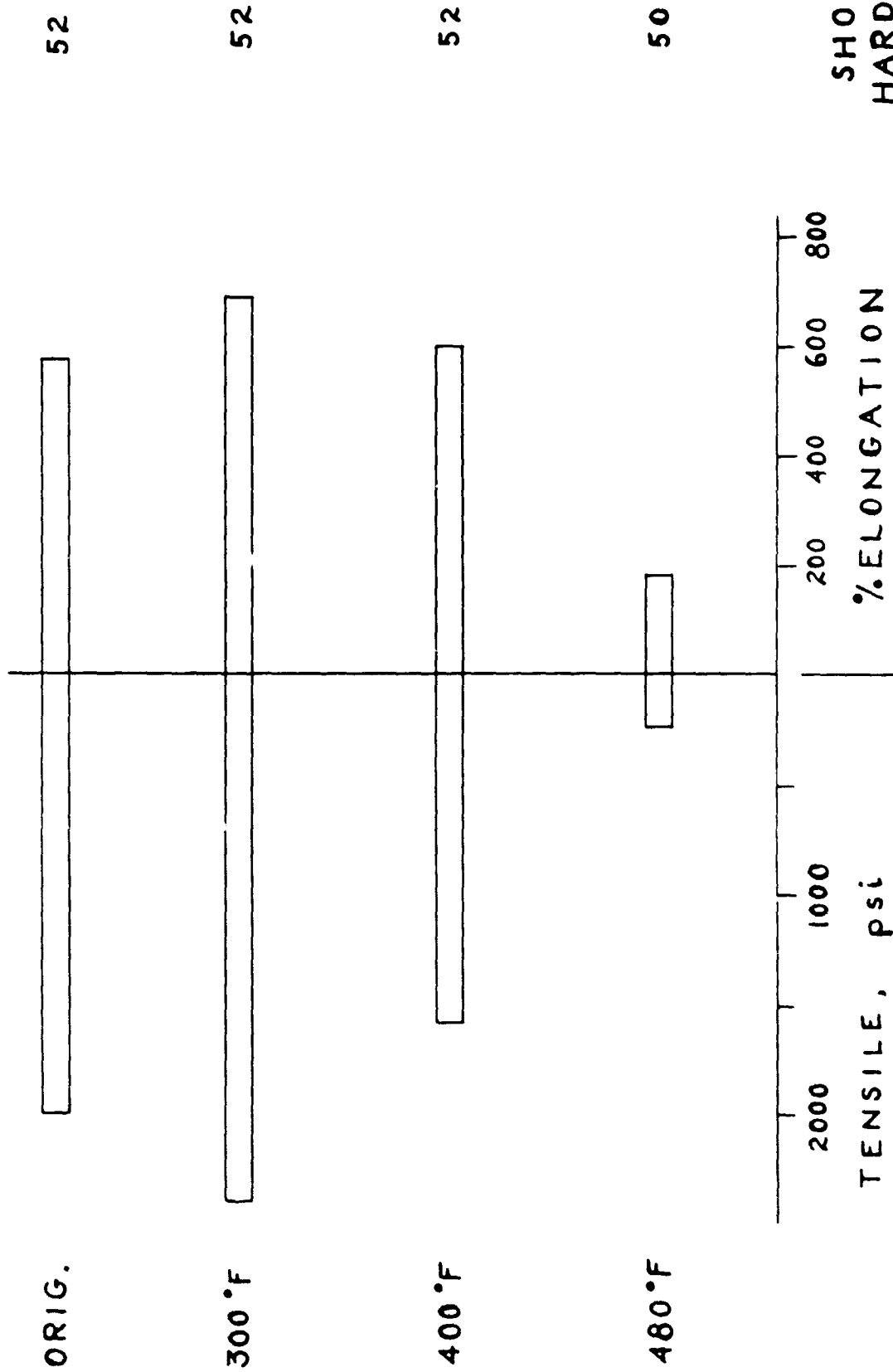
SLIDE VII

EFFECT OF HOT
OILS ON #89 VULCANIZATE

<u>MEDIA</u>	<u>ASTM #1 OIL</u>	<u>TEXAMATIC 1505 OIL</u>
<u>ORIGINAL PHYSICAL PROPERTIES</u>		
TENSILE STRENGTH PSI	1700	1700
PERCENT ELONGATION	630	630
SHORE A HARDNESS	56	56
PHYSICAL PROPERTIES AFTER 70 HOURS IMMERSION AT 300°F.		
PERCENT CHANGE IN TENSILE STRENGTH	+15.5	-5
PERCENT CHANGE IN ELONGATION	+25	+22
PERCENT CHANGE IN HARDNESS	0	-2
PERCENT VOLUME SWELL	0	+5

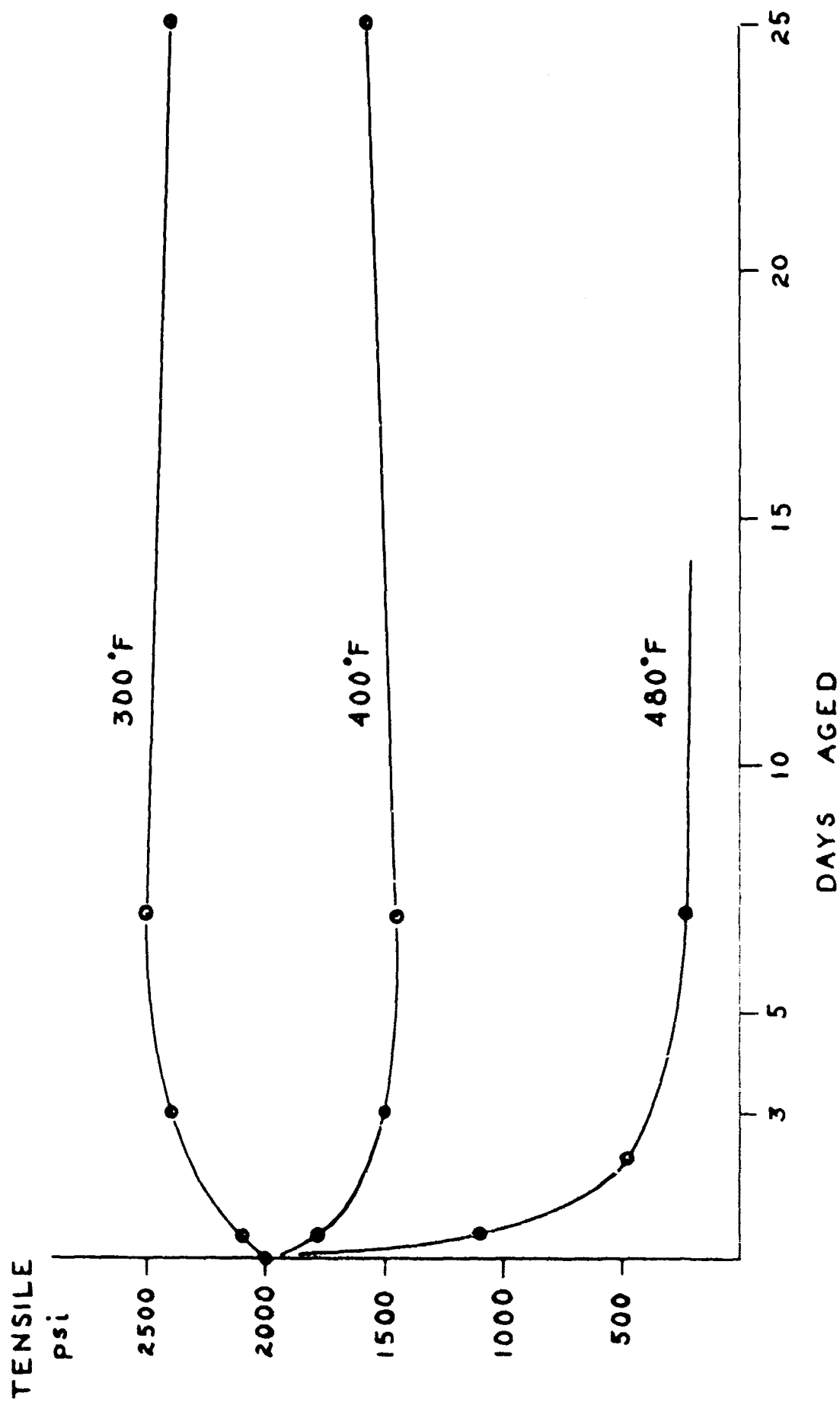
THERMAL STABILITY OF PEROXIDE CURED KEL-F Elastomer

30 DAYS IN CIRCULATING AIR OVEN



Slide VIII

THERMAL STABILITY OF PEROXIDE CURED KEL-F ELASTOMER



Slide II

SLIDE X

LOW TEMPERATURE PROPERTIES
KEL-F ELASTOMER

BRITTLE POINT	-51 C.
GEHMAN STIFFNESS	
T_2	-4°C.
T_5	-13
T_{10}	-17
T_{100}	-35

SLIDE XI

**AGING CHARACTERISTICS
PEROXIDE CURED KEL-F ELASTOMER**

OXYGEN BOMB:	No change in physicals or appearance; 7 days, 300 psig., 75°F.
OZONE:	No change in physicals, no surface defects; 6 hrs., 130 ppm., 30% stretch.
ULTRAVIOLET:	Slight increase in tensile, no change in elongation, hardness; 100 hrs., Fadometer.
WEATHERING:	No change in physicals; one year outdoor exposure, industrial atmosphere.

SLIDE XI

WATER ABSORPTION PROPERTIES OF
VARIOUS RUBBERS

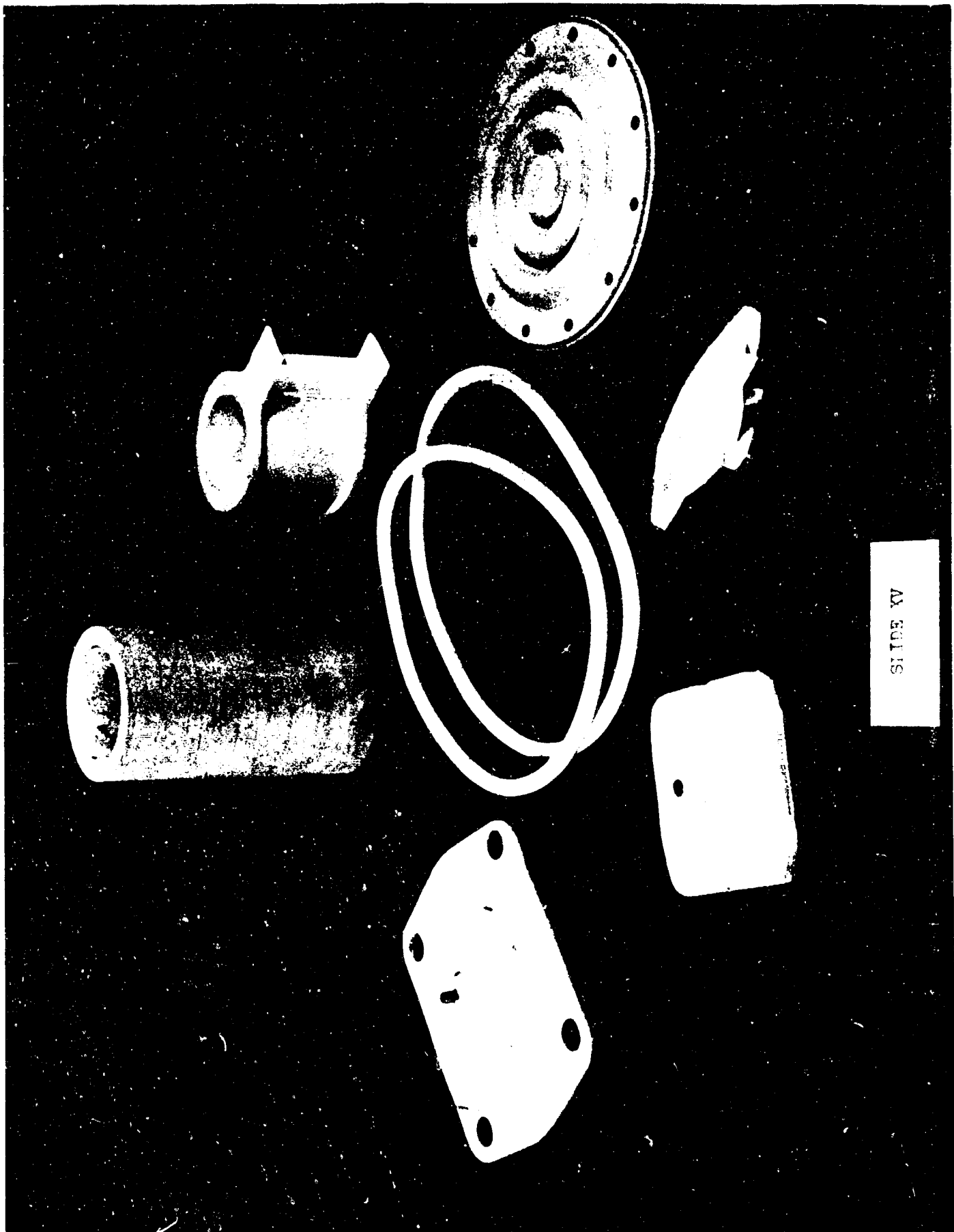
<u>RUBBER</u>	<u>DAYS</u>	<u>Mo. $\frac{g}{cm^2}$ at 77°F.</u>
SMOKED SHEET	58	12.32
NEOPRENE	58	12.9
DE PROTEINIZED RUBBER	63	6.38
GAS (elec. grade)	63	6.02
GRN	63	13.55
CHLOROSULFONATED POLYETHYLENE	63	5.69
NITRILE ELASTOMER (PVC)	63	3.5

SLIDE XIII

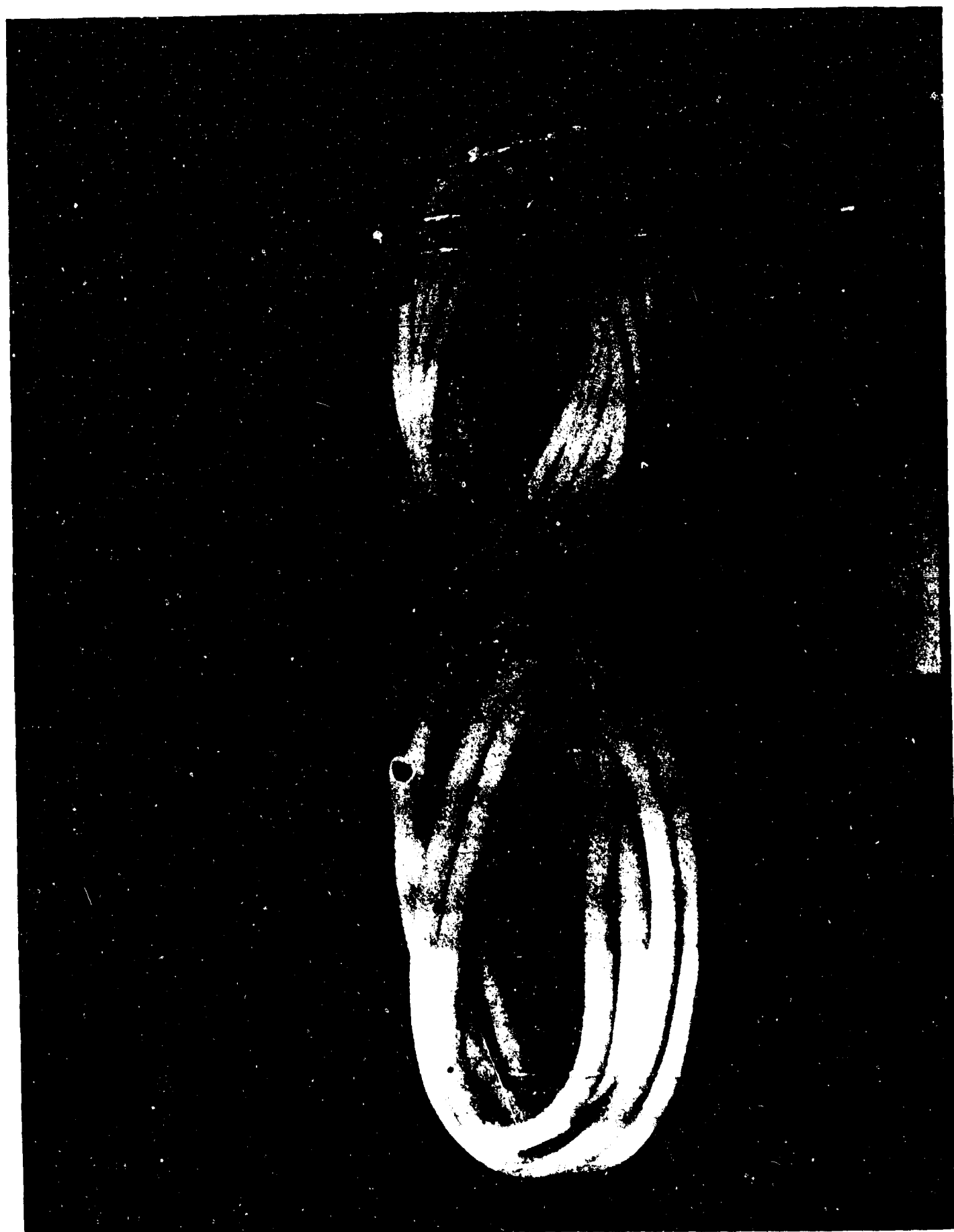
ELECTRICAL PROPERTIES OF KEL-F ELASTOMER

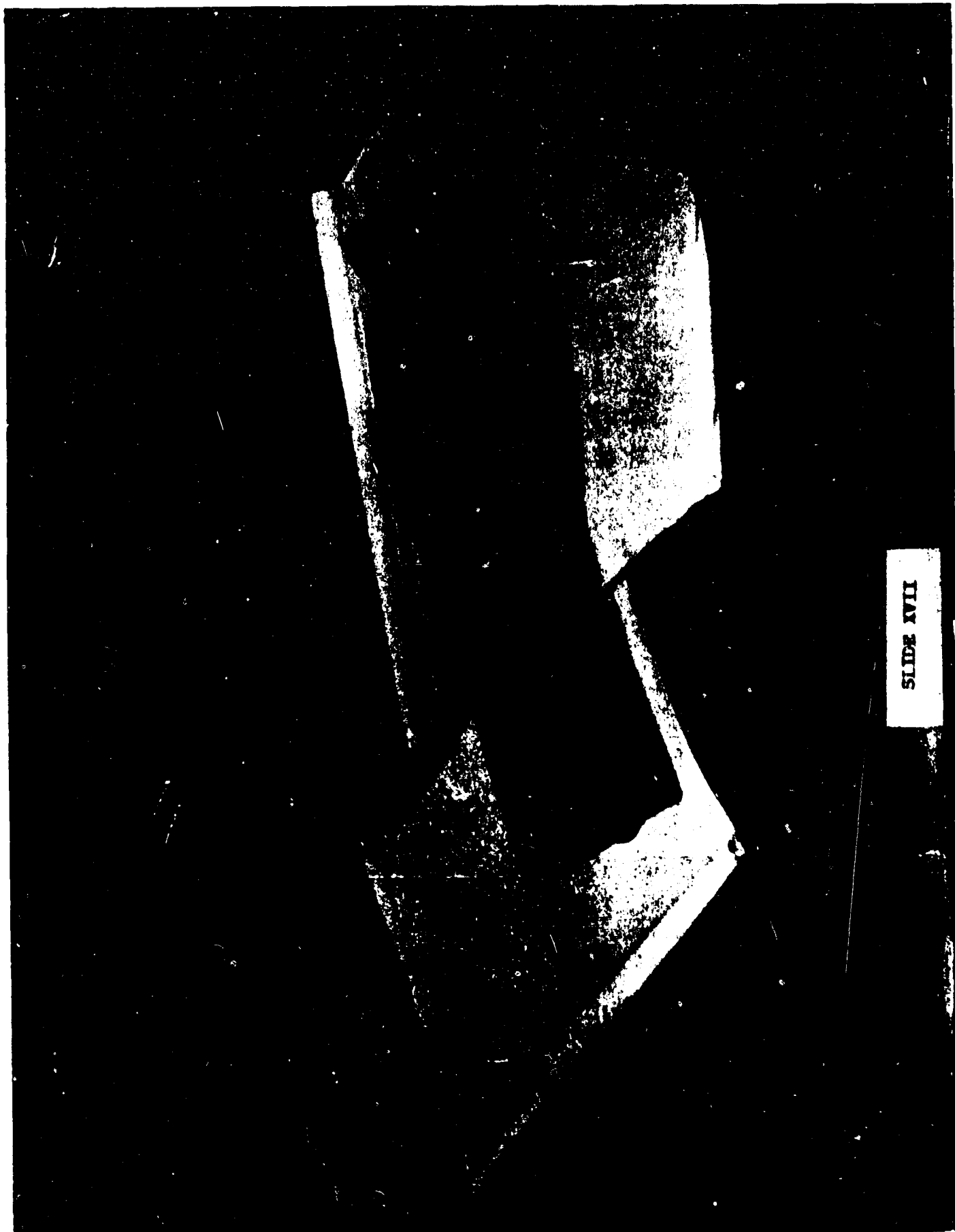
	<u>ORIGINAL</u>	<u>1 WEEK, 95% R.H., 23°C</u>
DIELECTRIC STRENGTH VOLTS/MIL	613	642
VOLUME RESISTIVITY OHM-CM	1.13×10^{14}	1.2×10^{14}
DIELECTRIC CONSTANT		
60 CYCLES	6.27	6.52
1000 CYCLES	5.90	6.08
10^6 CYCLES	3.94	4.05
POWER FACTOR		
60 CYCLES	0.025	0.028
1000 CYCLES	0.053	0.057
10^6 CYCLES	0.113	0.120





SLIDE W





SLIDE XVII